

WHAT IS CLAIMED IS:

1 1. A method for forming a dense composite of silicon nitride and silicon
2 carbide, said method comprising:

3 (a) mechanically activating a powder mixture of amorphous silicon nitride
4 and silicon carbide in the presence of at most 1% by weight of metal oxide
5 densification aids, said powder mixture consisting essentially of particles less than
6 100 nanometers in diameter; and

7 (b) consolidating said powder mixture into a continuous mass by compressing
8 said powder mixture while passing an electric current through said powder mixture, to
9 achieve a fused mass of silicon nitride and silicon carbide crystals.

1 2. The method of claim 1 in which said mechanically activated powder
2 mixture resulting from step (a) consists essentially of particles of about 1 micron to about 10
3 microns in diameter, and said fused mass produced in step (b) consists essentially of
4 crystalline grains less than 100 nm in diameter.

1 3. The method of claim 1 in which said mechanically activated powder
2 mixture resulting from step (a) consists essentially of particles of about 1 micron to about 5
3 microns in diameter, and said fused mass produced in step (b) consists essentially of
4 crystalline grains less than 50 nm in diameter.

1 4. The method of claim 1 in which any metal oxide densification aid
2 present in said powder mixture constitutes at most about 0.5% by weight of said powder
3 mixture.

1 5. The method of claim 1 in which any metal oxide densification aid
2 present in said powder mixture constitutes at most about 0.1% by weight of said powder
3 mixture.

1 6. The method of claim 1 in which said powder mixture is devoid of
2 metal oxide densification aids.

1 7. The method of claim 1 in which said powder mixture consists
2 essentially of from about 10 to about 60 parts by volume silicon, from about 10 to about 60

3 parts by volume carbon, and from about 10 to about 60 parts by volume nitrogen, based on a
4 total of 100 parts by volume of said powder mixture.

1 8. The method of claim 1 in which said powder mixture consists
2 essentially of from about 10 to about 30 parts by volume silicon, from about 25 to about 50
3 parts by volume carbon, and from about 25 to about 50 parts by volume nitrogen, based on a
4 total of 100 parts by volume of said powder mixture.

1 9. The method of claim 1 further comprising forming said powder
2 mixture by pyrolysis of a polyorganosilazane in an inert atmosphere.

1 10. The method of claim 9 in which said polyorganosilazane is a
2 polyureasilazane.

1 11. The method of claim 1 in which step (b) comprises compressing said
2 powder mixture at a pressure of about 10 MPa to about 200 MPa and a temperature of from
3 about 900°C to about 3,000°C, and said electric current is a pulsed direct current of about
4 1,000 A/cm² to about 10,000 A/cm².

1 12. The method of claim 11 in which said pressure is about 40 MPa to
2 about 100 MPa.

1 13. The method of claim 11 in which said temperature is about 1,000°C to
2 about 2,000°C.

1 14. The method of claim 11 in which said pulsed direct current is about
2 1,500 A/cm² to about 5,000 A/cm².

1 15. The method of claim 1 in which step (b) is performed to achieve a
2 fused mass with a density of at least 95% relative to a volume-averaged theoretical density.

1 16. The method of claim 1 in which step (b) is performed to achieve a
2 fused mass with a density of at least 98% relative to a volume-averaged theoretical density.

1 17. The method of claim 1 in which step (b) is performed to achieve a
2 fused mass with a density of at least 99% relative to a volume-averaged theoretical density.

1 **18.** The method of claim 1 in which step (a) comprises milling said powder
2 mixture by high-energy ball milling.

1 **19.** The method of claim 18 in which said high-energy ball milling is
2 performed with silicon nitride milling balls in a rotary mill at about 6 impacts per second or
3 more and a charge ratio of at least about 10.

1 **20.** The method of claim 18 in which said high-energy ball milling is
2 performed with silicon nitride milling balls in a rotary mill at from about 6 to about 60
3 impacts per second and a charge ratio of about 10 to about 20.

1 **21.** A dense composite of silicon nitride and silicon carbide consisting
2 essentially of silicon nitride crystals of less than 100 nanometers in diameter and said silicon
3 carbide crystals of less than 100 nanometers in diameter and containing at most 1% by weight
4 of metal oxide densification aids, produced by a process comprising:

5 (a) mechanically activating a powder mixture of amorphous silicon nitride
6 and silicon carbide in the presence of at most 1% by weight of metal oxide
7 densification aids, said powder mixture consisting essentially of particles less than
8 100 nanometers in diameter; and

9 (b) consolidating said powder mixture into a continuous mass by compressing
10 said powder mixture while passing an electric current through said powder mixture, to
11 achieve a fused mass of silicon nitride and silicon carbide crystals.

1 **22.** The dense composite of claim 21 in which said mechanically activated
2 powder mixture resulting from step (a) consists essentially of particles of about 1 micron to
3 about 10 microns in diameter, and said fused mass produced in step (b) consists essentially of
4 crystalline grains less than 100 nm in diameter.

1 **23.** The dense composite of claim 21 in which said mechanically activated
2 powder mixture resulting from step (a) consists essentially of particles of about 1 micron to
3 about 5 microns in diameter, and said fused mass produced in step (b) consists essentially of
4 crystalline grains less than 50 nm in diameter.

1 **24.** The composite of claim **21** in which any metal oxide densification aid
2 present in said powder mixture constitutes at most about 0.5% by weight of said powder
3 mixture.

1 **25.** The composite of claim **21** in which any metal oxide densification aid
2 present in said powder mixture constitutes at most about 0.1% by weight of said powder
3 mixture.

1 **26.** The composite of claim **21** in which said powder mixture is devoid of
2 metal oxide densification aids.

1 **27.** The composite of claim **21** in which said powder mixture consists
2 essentially of from about 10 to about 60 parts by volume silicon, from about 10 to about 60
3 parts by volume carbon, and from about 10 to about 60 parts by volume nitrogen, based on a
4 total of 100 parts by volume of said powder mixture.

1 **28.** The composite of claim **21** said powder mixture consists essentially of
2 from about 10 to about 30 parts by volume silicon, from about 25 to about 50 parts by
3 volume carbon, and from about 25 to about 50 parts by volume nitrogen, based on a total of
4 100 parts by volume of said powder mixture.

1 **29.** The composite of claim **21** in which said powder mixture is formed by
2 pyrolysis of a polyorganosilazane in an inert atmosphere.

1 **30.** The composite of claim **29** in which said polyorganosilazane is a
2 polyureasilazane.

1 **31.** The composite of claim **21** in which step (b) comprises compressing
2 said powder mixture at a pressure of about 10 MPa to about 200 MPa and a temperature of
3 from about 900°C to about 3,000°C, and said electric current is a pulsed direct current of
4 about 1,000 A/cm² to about 10,000 A/cm².

1 **32.** The composite of claim **31** in which said pressure is about 40 MPa to
2 about 100 MPa.

1 **33.** The composite of claim **31** in which said temperature is about 1,000°C
2 to about 2,000°C.

1 **34.** The composite of claim **31** in which said pulsed direct current is about
2 1,500 A/cm² to about 5,000 A/cm².

1 **35.** The composite of claim **21** in which said fused mass has a density of at
2 least 95% relative to a volume-averaged theoretical density.

1 **36.** The composite of claim **21** in which said fused mass has a density of at
2 least 98% relative to a volume-averaged theoretical density.

1 **37.** The composite of claim **21** in which said fused mass has a density of at
2 least 99% relative to a volume-averaged theoretical density.

1 **38.** The composite of claim **21** in which step (a) comprises milling said
2 powder mixture by high-energy ball milling.

1 **39.** The composite of claim **38** in which said high-energy ball milling is
2 performed with silicon nitride milling balls in a rotary mill at about 6 impacts per second or
3 more and a charge ratio of at least about 10.

1 **40.** The composite of claim **38** in which said high-energy ball milling is
2 performed with silicon nitride milling balls in a rotary mill at about 6 to about 60 impacts per
3 second and a charge ratio of about 10 to about 20.